EFFECT OF DISTRIBUTION COEFFICIENTS: SILICA SOL BONDED HIGH ALUMINA CASTABLES

A
THESIS IN THE PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF
MASTER OF TECHNOLOGY

BY

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NATIONAL INSTITUTE OF TECHNOLOGY
ROURKELA

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CERTIFICATE

This is to certify that the thesis entitled, "**EFFECT OF DISTRIBUTION COEFFICIENTS: SILICA SOL BONDED HIGH ALUMINACASTABLES**", submitted by **Mr.Arun Kumar** student under **Dr. Ritwik sarkar** carried out in OCL India Ltd, Rajgangpur, in partialfulfillment of the requirements for the award of Master of Technology Degree in **Ceramic Engineering** at the National Institute of Technology, Rourkela is anauthentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the results embodied in this thesis have not been submitted to any other University/ Institute for the award of any degree or diploma.

I wish him all the success for his future.

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30/5/14

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CONTENTS

ABSTRACT

Refractories used in various areas of the iron and steel industry need to face severe challenges to come across critical operational parameters at high temperatures. Hence, refractory manufacturers tend to shift towards unshaped refractories to substitute for conventional and shaped ones. Castables is uppermost in all the areas among the different unshaped refractories. Refractory castables (which are used by casting method) are premixed combinations of refractory grain, matrix components, bonding agents, and additives. The proportions are varied accordingly to attain the desired properties. Flowability of the castables is very important for application (casting) purposes and their final performances. Better flow of any castable allows making a good lining and makes it possible to make an intricate shape. This flowability is dependent on the particle size distribution and packing of the castable system. But better flowing mass may indicate higher sphericity of particles, resulting in lower strength, which is not acceptable for refractory application. Hence design of refractory castable system is challenging as it required opposing character of flowability with higher strengths. The present work targets to study the development of high alumina silica bonded refractory castable system with particle size distribution (PSD) as per well-known Dinger Funk model for continuous distribution and study the developed properties of the castables for their flowability and various characteristics developed at different temperatures.

CHAPTER 1

INTRODUCTION

INTRODUCTION

Ceramic technology is the earliest technology; more than 24000 years old and at the same time is the most modern, dynamically developing and diversifying field. The ever-increasing uses of ceramics in different areas of space age technology have made it a field of planned importance. Amongst the different classes of ceramics, refractories are the materials having those chemical and physical properties that make them applicable for structures or as components of system that are exposed to environments above 1000^{o}F [as per ASTM, 1984]. Hence refractories are the important component for all high temperature processing. Refractories need to have heat with stand ability, strength at higher temperatures, resistance to corrosive atmosphere as per application [1, 2].

Refractories are that group of ceramic materials which are used to line container in which other materials like metals; glass and cements are manufactured at higher temperatures. They include of discontinuous large sized aggregate phases along with continuous finer binder phases in the matrix. They are mainly separated as shaped and unshaped (Monolithics) refractories [3].

Refractories application in various industries like:

- Iron & Steel Industry
- Cement Industry
- Aluminium Industry
- Tile Industry
- Petro Chemical Industries
- Oil Industries
- Glass Industries

In the last two decades the use of monolithic or unshaped refractories has been leading over the preshaped refractories. It has been observed that the ratio of monolithics to preshaped refractories has been increasing gradually because of the dramatic decrease in the consumption of the shaped ones [3].

“Monolithics” has come from words “mono” that meaning single and “lithus” meaning stone/structure. It includes various material and compositions, along with a wide range of bonding systems. Monolithics are single piece cast which takes the shape of equipment. Monolithics have emerged into a useful class of refractory materials offering performance and cost-effectiveness superior to those of shaped ones. The success of monolithics is, due to the significant advancements in the type and quality of their binders, aggregates, and admixtures in addition to their design creation and innovative installation techniques [3].

Monolithics have quickly grown at the expense of bricks because of their faster, easier and cheaper installation; ready availability and minor corrosion liable lining joints, greater volume stability and better spalling resistance [4].

The iron and steel industry has undergone innovatory changes because of the adaptation of various classy processes to improve the quality and production of steel. The changes have been impressive. As a significance, refractory materials used in various areas of the iron and steel industry need to face severe challenges to meet critical operational parameters at high temperature. Refractories should perform better with less down time, as has been forced by steel manufacturers. Hence, the attention of refractory researchers, manufacturers and users has

removed towards unshaped refractories to substitute for the conventional and shaped ones. Among the different unshaped refractories, castables lead in all areas including Research & Development, as well as in manufacturing and application areas. Physical, mechanical, chemical and thermo-mechanical properties of various castables as well as their processing and bonding mechanisms are usually considered [1].

The most significant advancement in monolithics technology was the development of castables based on silica sol binder. Refractory castables are pre mixed combinations of refractory grain, matrix components, bonding agents, and additives. The proportions of each component used vary in each castable composition to attain the desired properties essentially required for the intended application. The curing and dewatering steps must be controlled by carefully to prevent explosive spalling. The material is mostly provided dry but sometimes installed by ramming or vibro-casting along with the addition of proper amount of water or other liquid, which helps as the binder.

So ever-increasing demand and application of unshaped refractories to replace conventionally shaped refractories due to major advantages which are (1) have inspired scientists and manufacturers to continuouse investigation of these materials in depth and to improve their quality and performance. Since the early 1960s, the availability of purer calcium aluminate cements and some other binder with higher alumina content has made the unshaped refractory system purer and allowed them to be applicable for various high-temperature applications (2). Among the various unshaped refractories, castables lead in all the areas of research fields, development, manufacturing and application. Physical, mechanical, chemical and thermo-

mechanical characteristics of various castable systems and their processing and bonding mechanisms too are the focus of such investigations.

The classification of castables in accordance to IS: 10570 are as follows:

1. Conventional – A castable containing greater than 2.5% calcium oxide on calcined basis.
    a. Dense castable
    b. Insulating castable – B.D having a maximum of 1.85 g/cc

2. Low cement castable – a castable containing calcium oxide greater than 1% and maximum of 2.5% on a calcined basis.

3. Ultra low cement – a castable containing calcium oxide greater than 0.2% and maximum of 1% on a calcined basis.

4. No cement castable – a castable containing calcium maximum of 0.2% on a calcined basis.

5. Gunning material – This material can be in the category of either conventional or low cement depending on its calcium oxide content. The gradation, based on calcium oxide content, is similar to that of castable. The installation of this product is done by a gunning machine.

While conventional castables containing the largest amount of cement make up the greatest percentage of those produced, use of reduced cement varieties like low cement castables

(LCC's) and ultralow cement castables (ULCC's), have grown significantly over the last decade. This is due to the CaO present in the cement which leads to decline in the high-temperature properties. Conventional castables consist 15-30% CAC, the amount being necessary to achieve satisfactory strength at low and intermediate temperatures. A comparatively large amount of water (0-5%) is taken up by the porosity of the aggregates which does not contribute to the hydraulic bond. The disadvantage of the conventional castables is closely related to the amount of cement present. The conversion of the bond phase between 250-350°C decreases the mechanical strength. The hot strength reaches a maximum value at 800-1000°C because of the formation of a ceramic bond. At elevated temperatures, the strength of silica containing castables decreases due to liquid formation. The hot strength can be improved by minimising the cement content of the castable [3].

The performance of alumina based castables has improved because of the reduction in the cement-content, thus diminishing the liquid phase formation at application temperatures and improving the corrosion and creep resistance. Thus low cement castables (LCC), ultra-low cement castables (ULCC), no cement castables (NCC) and self-flow castables (SFC) have popularised in the refractory industries [1],[3],[4]. Self-flow castables contain a greater amount of fines content while castables with lesser fines content require vibration to take shape. The amount of fines content in a castable is decided by the distribution coefficients. If it is less, then the castable has greater amount of finer particles. Usually the castable is self-flowing in nature if the distribution coefficient is less than 0.25 and vibratable if the distribution coefficient is more than 0.25 [6]. Self-flow castables flow under their own weight while vibratable castables require an external load. The binding phase used here is the high alumina cement (HAC).The major

phases present are $CaO.Al_2O_3$ (CA), $CaO.2Al_2O_3$ (CA_2) and $12CaO.7Al_2O_3$ ($C_{12}A_7$). CA_2 phase is the slowest setting phase and $C_{12}A_7$ phase is the quickest setting phase. The setting time depends on the amount of CaO present in the phase. The higher the amount present, the more quick setting it is. The cements, mostly used as binders, mainly consist of CA_2 and CA phases [1,5,6]. The binding phase and the fired temperature differentiate the castables. After preparation, drying and firing takes place. The physical properties are then tested to see which binder composition and firing temperature gives the best properties. Again flowability of the castables is very important for application (casting) purposes and their final performances. Better flow of any castable allows making a good lining and makes it possible too line an intricate shape. This flowability is dependent on the particle size distribution and packing of the castable system. But better flowing mass may indicate higher sphericity of particles, resulting lower strength, which is not acceptable for refractory application. Hence design of refractory castable system is challenging as it required opposing character of flowability with higher strengths.

One of the points are still in the process of development in these systems is the need for binders with greater refractoriness and less sensitive to conditions of cure that traditionally employed cements calcium aluminate (CAC)[7].). For the system of Al-CaO-SiO , additions greater than 2 to 2-3% by weight of CAC may induce the formation of phases ones of low melting point (1200-1300 (C) .[7-8]

Colloidal silica's (CS) are stable water based suspensions that, when combined with castable particles, provide, among other advantages, a higher drying speed and sinterability for the product.[9] Colloidal silica sols are stable water-based suspensions, containing up to 50 wt% of nanometric

spherical amorphous silica particles (8-15 nm diameter). These materials have been broadly used in the formulations of coatings, catalysts, papers, and anti-soil treatments as well as high temperature binders for fibrous isolating and investment castings [10, 11]. When combined with other solid particles, colloidal silica can be linked together in branched chains, in a process known as gelation [12, 13], which can be induced by water removal. During the drying step, the hydroxyl groups (Si-OH) on the surface of the particles generate siloxane bonds (Si-O-Si), which results in a three-dimensional network.

Gelation can also be induced by pH changes and a salt or water-miscible organic solvent addition, named gelling agents. With the proper selection of these agents or the pH variation, the colloid is gelled around the solid particles providing mechanical strength to the system after drying [12, 13-14]. The resulting structure is highly permeable and allows the composition to be easily dried, reducing cracks and explosive spalling. During sintering, its high surface area (typically close to 200 m /g) may induce the formation of mullite in alumina-based system, which improves the chemical resistance and the hot-strength properties [14].

CHAPTER 2

LITERATURE REVIEW

LITERATURE REVIEW

2.1 REFRACTORIES:

Refractories are those inorganic nonmetallic materials which can withstand high temperature without undergoing physico–chemical changes while remaining in contact with molten slag, metal and gases at high load even under the utmost conditions of highly corrosive and abrasive environments. As per ASTM C-71 refractories are materials having those physical and chemical properties that make them suitable for structures exposed to temperatures above 1000°F.

Refractories are classified on the basis of chemical nature, shape, porosity, carbon percentage, heat withstanding capability, and purity. On the shape basis, refractories are further divided into shaped and unshaped refractories.

First type or Shaped type refractories are manufactured by pressing while unshaped ones (monolithics) are either self-flowing or vibratable in their types [15-18].

Monolithic refractories are replacing conventional brick refractories in steelmaking and other metal removal industries. Monolithic refractories usually are loose materials that can be used to form joint free lining. Some of the main advantages of monolithic linings are

- Grater volume stability
- Better spalling tendency
- Complete elimination of joint in comparison with brick lining
- Can be installed in hot standby mode

- Transportion is easier

Monolithic refractories is installed by casting, spraying etc. Ramming masses are used typically in cold condition so that desired shapes can be obtained with accuracy.

2.2 Characterization of the products

The following properties of the samples were measured for samples that are dried and fired at different temperatures.

2.2.1 Bulk Density

BD is the just ratio of the mass of the dry material of a porous body to its bulk volume expressed in gm/cm^3 or kg/m^3, where bulk volume is the sum of the volumes of the solid material, the open pores and the closed pores in a porous body.

The weight of the dried sample is taken and its dimension is measured with the help of vernier callipers and its volume (length x breadth x height) is calculated. Bulk density is then calculated by dividing weight by volume.

Bulk Density=weight/ (length x breadth x height) (units gm. /cc) The same is done for the fired samples. Or another method by which it can be measured

Whereas true density is the ratio of mass of the material of a porous body to its true volume and true volume is the volume of solid material in a porous body.

B.D = (W_1/W_3-W_2) × density of liquid at temperature of test

(e.g. density of water at 25°C : 0.997044 gm/cc, at 30°C : 0.995646 gm/cc)

2.2.2Cold Crushing Strength

The cold crushing strength of the samples is measured by a compressive Tester. Cold crushing strength is defined as the maximum amount of load the sample can withstand before complete breakage .Cold crushing strength is given by the formula load/area. Cold crushing strength of the refractories is measured by placing a suitable refractory specimen of on flat surface followed by application of uniform load to it through a bearing block in a standard mechanical or hydraulic compression machine of testing. The quantity load at which crack appears in the refractory specimen represents the cold crushing strength of specimen. The load is applied evenly on the sample in the flat position. It is expressed as kg/cm^2 (Units kg/cm^2) Load at which fracture occurs in kN, Area of the Samples in cm^2

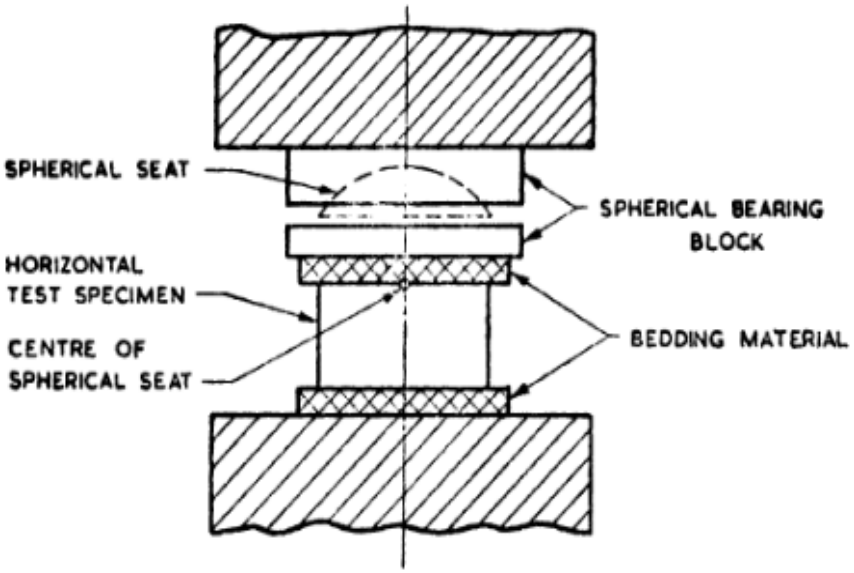


Fig: schematic diagram of CCS

2.2.3Permanent Linear change (PLC) on reheating:

In materials certain permanent changes occur during heating and these changes occur due to; change in the allotropic form, chemical reaction, liquid phase formation, sintering reactions

PLC (%) linear = (Increase or decrease in length/ original length) × 100

With the help of these changes determined the volume stability, expansion and shrinkage of the refractory at high temperatures.

2.2.4Flowability Test

The flow value of the castables is found out using an ASTM C 230 flow cup of height 5 cm and having a base of base 10 cm. After passing 30 seconds the flow value is measured by measuring the change in the diameter of the base with the help of a ruler and the values are reported in mm.

2.3 MONOLITHICS:

Monolithic refractory, the name commonly given to all unshaped refractory products, are materials installed as a form of suspension that ultimately harden to form a solid mass. Most monolithic formulations consist of large refractory particulates (an aggregate), small filler materials (which fill the interparticle voids) and a binder phase (that gels the particulates together in the green state). They are already mixed combinations comprising of refractory grains as the aggregate (coarse) phase, binder which works as the matrix phase, and other additives. These materials finally harden on mixing with water or liquid, to form a solid mass. The reasons

for the success of monolithics, above shaped bricks, are their rapid availability, easier and cheaper installation, and minor corrosion susceptible lining joints, greater spalling resistance, and better volume stability [19,20].

2.3.1Types of Monolithic Refractory

CASTABLE REFRACTORIES

These are materials which consist of precision graded coarse and fine refractory gr ai ns. They are gelled by means of a binder system in the materials unfired state. After the heat-up of the material the binder either transforms or volatilises facilitating the formation of a ceramic sintered bond. The mostly used binder in castables is HAC (high alumina cement). Some other binders that are often used comprise hydratable aluminas and colloidal silica. First of all wateris madded and Castables is mixed with water and then installed by either pouring or pumping. then vibration is required for placement of the material.

The cement-containing castables are often classified by the amount of cement added to them. Conventional castables may contain around 15- 30% c ement binder. As refractory technology evolved chemical additives were included in the package to reduce the amount of cement and water the product required -the impact of this was material with improved strengths and stability. Low cement castables contains about 3- 10% weight cement .Ultra low cement castables contain less than 3% cement component.

A specialized type of refractory castable is the free flow castable which is able to be installed without any vibration. They require a lower amount of water addition than old-style castables .

This is only due to the fact that they contain particle packing and dispersing agents which modify the surface chemistry of the fine particles to improve the flow of the material.

Certain castable formulations may be installed via gunning techniques which involves spraying the material through a nozzle at a great speed. At nozzle, cement accelerators are usually added to promote rapid hardening of the material. By this technique applications can be lined very quickly.

PLASTIC REFRACTORIES

These are monolithic refractory materials which are tempered with water and/or added with a binder. They have enough plasticity to be pounded or rammed into place.

RAMMING REFRACTORIES

These materials are exact similar to plastic refractories though are much stiffer mixes.

PATCHING REFRACTORIES.

These materials are somewhat similar to plastic refractories though have a very soft plasticity allowing them to be pounded into place.

COATING REFRACTORIES

This type of product is used to protect refractory linings usually against chemical outbreak. Coating refractories are generally intended to cover just the working surface of a lining. They are likely to be fairly thin layers.

REFRACTORY MORTARS

Mortars consist of finely ground refractory materials which are then mixed with water to form a paste. Refractory products such as bricks can be laid and bonded by these materials. They are usually applied by trowelling.

INSULATING CASTABLES

Insulating castables are specialized monolithic refractories that are used on the cold face of applications. There are made from light weight aggregate materials such as vermiculite, perlite, extend-o-spheres, bubble alumina and expanded clay. Main function these light weight material is to bring thermal insulation. They are characteristically of low density and low thermal conductivity Insulating refractories have inferior mechanical strength to that of conventional castables.

2.4 CASTABLES:

Castable refractory refers refractory which is installed by casting. A significant advance in monolithics technology was the development of castables based on Calcium Aluminate Cements (CAC). Refractory castables include of graded refractory aggregates and hydraulically setting cement of calcium aluminate variety, with other additives for precise purposes. The material is generally supplied dry and might be installed with the appropriate addition of liquid or water, which serves as binder.

For casting refractory needs water to promote particles flowing and low viscosity. Water addition can be divided into three shares: a portion which fills the porosity of the raw materials, a portion which fills the voids between particles and the excess water which contributes to increase the interparticles distances. The two first ones don't contribute to the shaping process.during mixing and shaping, excess water allows to create a suspension and promotes the lubrification layer between particles is one of the most important parameters which determines the rheological behavior. A castable can only flow when the water addition exceeds the quantity required to fill

the whole volume of intra pores and interparticle voids. So it is obvious that optimization of the grain size distribution leads to decrease the quantity of water necessary for casting.[21]. But water requirement for cement based castable is about 5to 7% of weight.

On the basis of flow, castables are sub-divided into self-flow and vibratable castables. Self-flow refractories mechanically flow under their own weight to take the shape of the mould whereas vibratable refractories require vibration to take the shape of the mould [22, a, b, 23].

As calcium aluminate cement (CAC) based castable have improved now a days than before, but disadvantage of CAC bonded castable is presence of CaO phase which is low melting phase. The increasing application and demand for monolithic refractory materials as a substitute for conventional bricks encourage manufacturers and researchers to explore their physical, chemical and mechanical properties at elevated temperature as well as their processing techniques [24], [25] and [26]. A significant increase in the life of high-alumina based refractory castables has been possible by decreasing the cement-content, thus avoiding liquid phase formation in them at working conditions which in turn improves the corrosion resistance and creep strength. LCC, ULCC, NCC and self-flow castables have already become popular in the refractory industries [27], [28] and [29]. More recently, a new group of additives prepared from sol‑gel route, appeared in the refractory industries that triggered on a new prospect for castable producers. These additives comprise ultrafine particles of the same composition as the castable (e.g., silica, alumina, mullite, spinel etc.) which surround the refractory materials through a polymeric network skeleton and on heating, the reactive gel phases develop ceramic bonding and high strength through sintering [30]. These compositions have better flow properties and require

no vibration to help the material move, i.e., these castables render higher flexibility during the installation. These also help in decreasing the cement contents in castables thus reducing the amount of low melting gehlenite and anorthite phases.[31]

Great attempts were made to reduce the amount of calcium aluminate cement (CAC) content in refractory castables to improve their hot strength. If we use more than 2-3 wt% CAC may cause low melting phases formation in the refractory matrix leading to weak thermo-mechanical properties of the castables.

Colloidal silica bonded refractory castables have been developed recently. It was found that colloidal silica is one of the best binders can substitute other binders such as cement.
Colloidal Silica can affect the structure of refractory castables to achieve superior thermo mechanical properties. Replacing calcium aluminate cement (CAC) by colloidal silica as a water base binder, speeds up drying, reduces the amount of liquid phase at high temperatures and may lead to mullite formation, which increases the hot strength of the refractory castables. In this research, the influence of colloidal silica addition on bulk density, apparent porosity and HMOR of a tabular alumina based refractory castable containing have been studied . The results showed that samples containing colloidal silica have higher hot strength compared to those containing only CAC as binder due to the better compaction, less liquid phase formation at high temperature.

Silica and water content was kept constant. It's concluded that the castables containing the optimum amount of silica sol shows remarkable increase in both castable fluidity and mechanical

strength (CCS and MOR) in dried and fired state. It was found that nanosilica particles too increase the rate of needle-shaped mullite formation during sintering at 1400°C. According to FTIR results, the addition of Calcium Aluminate Cement (CAC) to the silica sol may be responsible for the increment of siloxane bridges (Si-O-Si)[32].

On the basis of overall results, it is concluded that the Optimum amount of silica sol is a major factor where it behaves as a binder. Specifically in refractory castable it should provide suitable mechanical strength, without significant reductionin fluidity. In the present study, 5% wt silica sol (like the MS4-NS2 sample) was known as the optimum content because it improved both fluidity and mechanical strength. By increasing silica sol, the castable fluidity begins to increase (like the sample containing 2.5% and 5%wt silica sol) and but it will be decreased dramatically in higher amounts (like the sample containing 7.5%wt silica sol). In addition, the simultaneous effect of gelation mechanism by optimum content of silica sol and the making of stratlingite phase have resulted in the increment of castable mechanical strength. It may be concluded that the mullite crystals couldn't grow appropriately during 3 hours sintering at 1400 ºC in the sample without silica sol. In this sample the mullite crystals could not be found by SEM evaluation, but the microstructural investigation reveals that the needle-shape mullite crystals were grown properly in samples containing nano silica by addition of silica sol. The amount of IR transmission for the silica sol containing CAC sample was higher than that for the pure silica sol (CS) one. It's related to the siloxane bond formation and gelling mechanism which could take place in the Ce-CS sample more than in the CS sample [32].

Colloidal silica sol could behave as a liquid and so the castables containing colloidal silica need less water to be prepared, which leads to lower their apparent porosity than that of LCC ones. Colloidal silica, due to its high specific area, expects sintering and so leads to better compaction. Comparing the traditional low calcium aluminate cement castables, colloidal silica containing refractory castables attains better mechanical strength at high temperatures [33].

It has been found by M. R. Ismael that the use of colloidal silica in refractory castable compositions can be advantageous in comparison with the refractory cement and the hydratable alumina [9]. Lower torques values were required during the mixing step and an earlier turning point was attained, compared to the other compositions analyzed.[33,9] The mechanical strength values as a function of curing time for the CS bonded system showed a lower sensitivity to curing conditions, as a shorter drying time was necessary to attain suitable properties. This effect can be technologically useful, as it provides a reduction on the full processing time. The gelling mechanism of colloidal silica containing composition generated a high-permeability structure, facilitated water removal, preventing spalling [9].

The nanometer-sized colloidal silica particles also improved the system's reactivity, increasing the castable sinterability, promoting mullite formation [9]. Nevertheless, the porosity level developed was too high. Although the colloidal silica is a nano sized system, the results obtained showed that their contribution to the packing was low. Therefore, superior mechanical strength values could be achieved with an adequate particle-size distribution, which would result in lower porosity and consequently in high-quality performance refractory castables [9].

Also The easiness of mixture and drying, the elevated permeability and the low sensitivity to the conditions of cure of the connected system with silica sol, they allowed a reduction in the total time of processing of castable A raised up surface area of sílica sol produced a bigger reactivity of the system, favoring it sinterability [9]. By Comparing both the analyzed systems, found that the sol silica as binding agent may present advantages in relation to concrete containing calcium aluminate cement [9].The easiness of mixing and drying, the high permeability and low susceptibility to conditions of curing system connected with silica sol, allowed a decline in the total time of processing of castable of high alumina silica sol generated better reactivity of the system, favoring the sintering [9].

2.5 PARTICLE SIZE DISTRIBUTION:

There are two types of particle packing in refractories namely discrete packing and continuous packing. And particle size distribution analysis has proven to be an effective tool in manufacturing and developing refractory castable. The type of packing plays a very crucial role in monolithics. To obtain higher density packing the principle consists in combining large and fine in such a way that finer particles fulfill the voids between the larger ones. For a bimodal grain size distribution, the higher density is obtained with a very high diameter ratio and a right proportion of the two families which allows to fill with smaller and smaller particles. Particle shape and surface roughness are also important parameters, equiaxed and smooth particles promote a high packing density. Commonly, for a refractory product, a high density is obtained with a wide continuous grain size distribution of commercially available aggregates and fins powders. And such a distribution is described by andreasen relationship. The discrete packing model was proposed by Furnas. He stated that the maximum packing density can be obtained

when the smaller sized particles fill the voids between the larger sized particles. Andreasen model presumed that particles could not be of a particular size rather they fall within a narrow size range.

The CPFT (cumulative percent finer than) equation according to Andreasen is given by:

$CPFT = (d/D)^{q} \times 100$

Where d represents the particle size for calculation of CPFT, D denotes the maximum particle size, and q denotes the distribution coefficient also known as q-value.

Here q values in range of 0.30 to 0.50 gives the highest packing density, higher q value means lower content of small particles, so it is used when matrix phase is of lower refractoriness, compared to that of large grains.

Andreasen's model did not assume any minimum particle size for calculating CPFT. Instead he assumed that the particles are infinitesimally small which was practically not possible. So the Andreasen's model was modified by Dinger Funk who assumed a certain minimum particle size for calculating CPFT thus making it more practical.

Thus the CPFT as according to the Dinger Funk model is following:

$$\mathbf{CPFT = ((D^{q} - D_{min}^{q})/ (D_{max}^{q} - D_{min}^{q})) \times 100}$$

Where D signifies the particle size for calculating CPFT, Dmin denotes the minimum particle size, Dmax denotes the maximum particle size, and q denotes the distribution coefficient.

As according to dinger & funk it can be concluded that "distribution with an exponent greater

than optimum pack in a scheme approaching a minimum nonzero porosity . in other words , regadless of the width of the distribution, porosity will never approach zero. The greater the value of exponent, the higher will be the limiting porosity. Distribution with exponent less than 0.37 approach zero porosity as the width of the distribution increases. As finer and finer particles are added to the distribution, porosity will decrease and approach to zero".[34]

The target is to reduce the water requirement to reduce porosity and increase density. The need for packing is that it decides the castable is vibratable / self-flow mass and optimum packing requires less water for placement. The distribution factor dictates the Particle Size Distribution and the flow behavior of the castable.

It was found that castables require a unique but contradictory character of flowability and strength. Higher packing pertains to densification which in turn increases strength but results in less flow due to friction. Therefore highest packing is not the only criterion. Thus the new packing model that is considered is the continuous particle size distribution. Packing should be optimum such that the castable requires minimal water for setting implying less porosity which will give a higher packing density. Thus the castable will have improved physical properties in terms of CCS (cold crushing strength) and HMOR (hot modulus of rupture).

Flowability is one of the most important characteristics of castables. Installation of castables depends on the flow properties and on how soon the flow is lost because of setting. The loss of flow always has been one of the main problems of refractory castables. Amongst various self-flow castables, the no cement type is technologically very important. Use of suitable binder

(such as a sol) in no-cement-type castables and more judicial adjustment of particle-size grading to provide greater grain-to-grain interlock packing for higher strength without affecting the flowability are necessary for such development.[35]

The flow characteristics of the castable (whether self-flowing or vibratable) is always determined by the distribution coefficients. The castable will be self-flowing in nature if „q‟ is less than 0.25 else it is vibratable in nature. Lesser the q-value than greater amount of fines in the batch involving greater flow ability. So, Self-flowing castables have to possess greater amount of fine particles in order to be self-flowing in nature.

As Bjorn Myhre found out that if the castable have the same particle size distribution and amount of water (in vol%) then similar flow values should be expected for a range of different castables. And also says that we will easily predict amount of water needed to obtain a given flow[36].

CHAPTER 3

OBJECTIVES AND PLAN OF THE WORK

OBJECTIVES AND PLAN OF THE WORK

As we see unshaped refractory is replacing shaped refractory in the refractory market. As it has many advantages over shaped one like it has no joints, easy handling, In unshaped, refractory catable demand is increasing continuously. So there had been done a good amount of work to improve the binder of the castable. As we know cement is used as binder in castable , but cement contains CaO in it which is a low temperature melting material so it decreases refractoriness of refractory castable. So it is tried to either reduce amount of CaO or cement in refractory castable. So as conventional castable is replaced by low cement castable and ultra-low cement castable is used so its refractoriness can be increased. So objective of this study was to know the effect of the distribution coefficient on the refractory castable using silica sol as binder replacing calcium aluminate cement.

In this study binder cement or calcium aluminate cement used in refractory castable is completely replaced by silica sol. And using this binder in high alumina refractory castable effect of distribution coefficient in dinger & funk formula is investigated along with and without using reactive alumina in it. One more thing which is investigated here is effect of fixing the amount of silica sol and adding water in castable up to appropriate flow ability.

Result of sample were analyzed and a suitable distribution coefficient is selected on which refractory castable shows good thermo-mechanical properties.

CHAPTER 4

EXPERIMENTAL PROCEDURE

EXPERIMENTAL PROCEDURE

4.1 Batch formulation

Coarse aggregates - White tabular alumina (WTA), of diffrant fractions which is shown in following table:1.

Fine aggregates - WTA fines/Reactive Alumina

Binder – silica sol, silica sol+ water

By keeping q = 0.21, 0.23, 0.27 & 0.29 we calculate the percentage of particles required in the various size ranges for a batches of 20 kg, 15 kg.

The raw materials were then sieved in order to keep the aggregate sizes in a particular size range.

Size fraction(in mm)	Raw Material
3 mm to 6 mm	White Tabular Alumina
2 mm to 3 mm	White Tabular Alumina
1 mm to 2 mm	White Tabular Alumina
0.6 mm to 01.18mm	White Tabular Alumina
0.3 mm to 0.6 mm	White Tabular Alumina
0.045 mm to 0.3 mm	White Tabular Alumina
0.0025 mm to 0.045 mm	White Tabular Alumina
Reactive Alumina	Reactive Alumina

3.1 Table showing the size fractions and the corresponding raw material

By keeping the distribution coefficients at 0.21, 0.23, 0.27, 0.29 we calculate the percentage of particles required in the various size ranges for batch formation and correspondingly the weights of the material required in the various size ranges are calculated for a batch of 20 kg. Four different composition were made as per the above mentioned q values using only WTA and without using any reactive alumina as fines and as binder only silica sol was used as binder in one set of experimentation and then in another set of experimentation fixed amout of silica sol was used with water.

Next, for all the different q values, 4 composition were made using a fixed quantity of reactive alumina 10wt% of the batch as fines, partially replacing the WTA fines. Here also a set of experimental was done using only silica sol as binder and also using a fixed amount of sol with water. The details of the compositions are shown in the following table. Batch formulations are based on the four different q values, calculated as per the CPFT values for each size fractions using the Dinger and Funk's equation.

Details of the batch composition of first 4 batches without reactive alumina are as follows

Size fraction of WTA (mm)	Raw material amount (%)			
	q=.21	q=.23	q=.27	q=.29
3 to 6	17.0	17.7	19.5	20.35
2 to 3	9.0	9.1	9.8	10.1
1 to 2	13.0	13.8	14.5	14.8
0.6 to 1.0	9.0	8.8	9.1	9.15
0.3 to 0.6	10.0	10.4	10.5	10.4
0.045 to 0.3	22.0	21.3	20.4	19.75
0.0025 to 0.045	20.0	18.9	16.5	15.45

Table: 3.2 Showing amount of WTA in % with four q values without reactive alumina

Above 4 composition mentioned in table were used to prepare castable were without any reactive alumina. In 4 of them were with only silica sol were added & another 4 composition 4% silica sol+ water were added till the proper consistence and flowability is achieved.

	Raw material amount (%)			
Size fraction (mm)	q=.21	q=.23	q=.27	q=.29
3 to 6 WTA	17	17.7	19.45	20.35
2 to 3 WTA	9	9.1	9.8	10.1
1 to 2 WTA	13	13.8	14.45	14.8
0.6 to 1.0 WTA	9	8.8	9.05	9.15
0.3 to 0.6 WTA	10	10.4	10.45	10.4
0.045 to 0.3 WTA	22	21.3	20.35	19.75
0.0025 to 0.045 WTA	10	8.9	6.45	5.45
Reactive alumina	10	10	10	10

Table: 3.7 Showing amount of WTA in % with four q values with reactive alumina

4.2 PREPARATION OF CASTABLE SAMPLE

4.2.1 Mixing

The coarse and the fine fractions of the batch were first dry mixed separately in the Hobart mixer. After proper mixing among themselves they are mixed together in the mixer. Silica sol is added till the proper consistency or flowability was achieved.

Figure 1 mixer for castable

4.2.2 Casting

Water is added until required flowability is reached. After proper homogenising in the planetary mixer, it is taken out and then poured in the iron moulds of size 50mm x 50mm x 50mm and bar moulds of 160mm x 40mm x 40mm which have already been lubricated by grease. For filling of the molds, self flowing compositions were allowed to flow on its own weight and for the vibratables, vibrating table is used for vibration and filling of the molds. Excess mix is taken out and the resultant mix is smoothened by a trowel. The moulds are kept in the normal atmosphere for 24 hours for setting.

4.2.3 Curing, Drying and Firing

On the next day of casting all the compositions were demolded and allowed for air drying for 24 h. After air drying all the compositions were placed in the oven and dried at 110oC for 24 h.

dimensions and weight of the oven dried samples were taken. Next all the compositions were fired at 950°C for 2 hours and at 1550°C for 2 hours soaking time. After firing again the dimensions and weight of the samples were measured. Next the dried and fired compositions were used for various characterization.

4.4 Characterization of the products

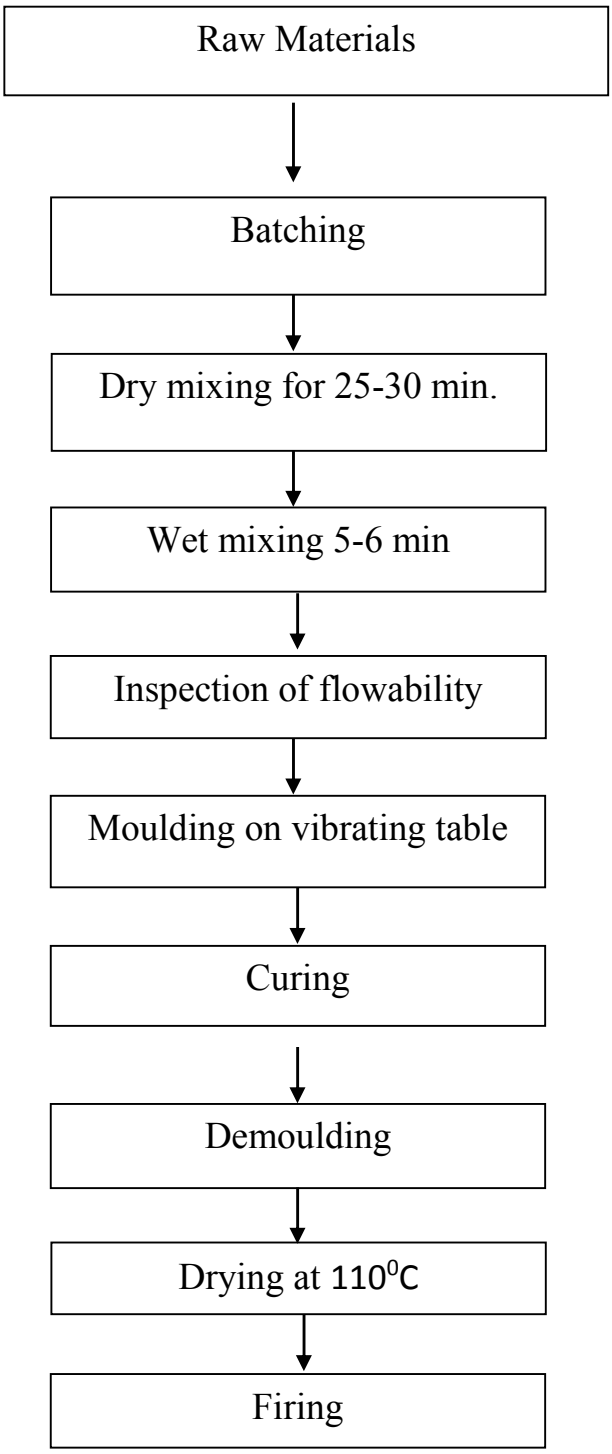
The following properties of the samples were measured for dried and different temperatures fired samples. Each data point represents an average value of three samples.

4.4.1 Flowability Test

The flow value of the castables is found out using an ASTM C 230 flow cup of height 5 cm and base 10 cm. First the mixed castables were placed in the flow cup and the the cup is drawn up allowing only the castable to remain and flow. The castable with self flowability will flow on its own weight and vabratables will flow after giving then a vibrational energy on vibrational table. After 30 seconds of flow, the flow value is measured by measuring the change in the diameter of the base with the help of a ruler and the values are reported as an average of 8 different directions in mm.

Figure 5: ASTM C-230 flow cup

4.3 Flow chart Preparation of samples



Flow chart:- castable sample forming processing

4.4.2 Bulk Density

The weight of the dried sample is taken and its dimension is measured with the help of vernier callipers and its volume (length x breadth x height) is calculated. Bulk density is then calculated by dividing weight by volume.

Bulk Density=weight/ (length x breadth x height) (units gm. /cc)
The same is done for all the fired samples.

4.4.3 Cold Crushing Strength

The cold crushing strength of the samples is measured by a compressive Tester. Cold crushing strength is defined as the maximum amount of load the sample can withstand before complete breakage .Cold crushing strength is given by the formula load/area. (Units kg/cm^2) Load at which fracture occurs in kN, area of the samples in cm^2.

Figure 2 UTM for CCS & MOR Test

4.4.3 Phase analysis by xrd:

The X-ray diffraction method was used for determining the phases in the castable fired at 1550 °C. The castable was crushed and grounded to very fine powders using a mortar pestle and then subjected to XRD for identification of the phases after sintering. XRD measurements are performed at a 3 °C/min scan rate using an X-ray diffractometer operated at 40 keV and 30 mA. The X- Ray data was plotted using the Expert Pro software and the peaks were matched with JCPDS software to get the phases present

4.4.4 Modulus Of Rupture

Modulus of rupture of a material indicates it's bending or tensile strength. A refractory sample is supported in span than load is applied at specific rate to the centre of the sample until sample breaks. The modulus of rupture is expressed in mega pascals (MPa). It is calculated or measured by load at which sample failed. If MOR is tested at room temperature than it is simply called MOR, and if it is measured at high temperature than called HMOR.
We can measure it By UTM.

CHAPTER 5

RESULTS & DISCUSSIONS

RESULTS & DISCUSSIONS

5.1 Densification study:

Increasing the q value was found to increase the density values due to greater extent of coarse content. Again increase in temperature to 900oc was found to deteriorate the density values for all the q values containing only sol or sol with water, may be due to the breaking of hydroxyl bond of sol and poor initiation of sintering, however further increase in temperature to 1550oC was found to enhance the densification strongly due to densification. Very fine silica particles from sol has resulted in good densification and produced high density at 1550oC. Compositions containing fixed sol and free water showed always a lower density values due to presence of excess water which results in voids after drying and also due to lesser presence of fine silica particles resulting in relatively poor densification at higher temperatures. Again addition of RA replacing WTA fines has resulted in marginally better density values, which may be associated with better flow properties and better packing in the compositions containing RA as fines.

q Value	BD (gm/cc)		
	110°C	900°C	1550°C
0.21	3.04	3.00	3.05
0.23	3.05	3.03	3.13
0.27	3.07	3.06	3.14
0.29	3.08	3.09	3.23

5.1 Density values of compositions containing only WTA fine and sol only

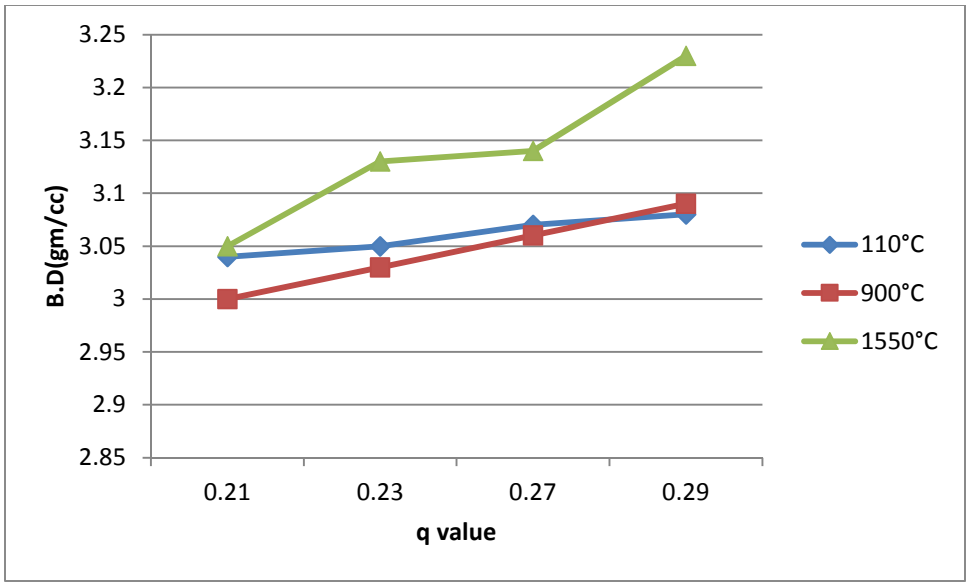


Figure 5.1:-Plot of Bulk Density vs. Q value of compositions containing only WTA fine and sol only

Q Value	BD (gm/cc)		
	110°C	900°C	1550°C
0.21	2.99	2.99	3.02
0.23	2.99	3.05	3.06
0.27	3.01	2.96	3.08
0.29	3.04	3.06	3.14

5.2 Density values of compositions containing only WTA fine and sol + water

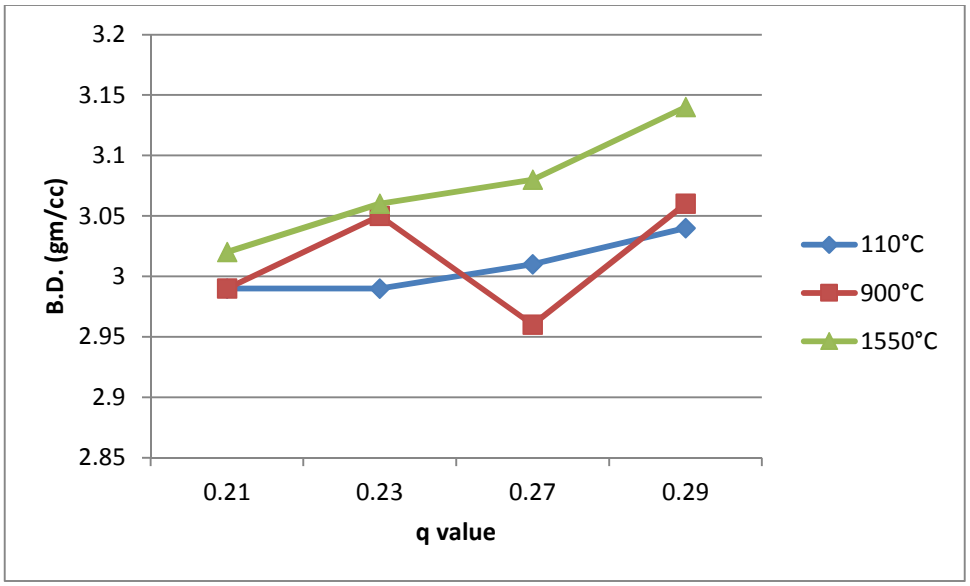


Figure 5.2:-Plot of Bulk Density vs. Q value of compositions containing only WTA fine and sol + water

Q Value	BD (gm/cc)		
	110°C	900°C	1550°C
0.21	3.06	3.01	3.09
0.23	3.08	3.06	3.13
0.27	3.09	3.10	3.18
0.29	3.13	3.13	3.25

5.3 Density values of compositions containing only WTA fine and RA and sol only

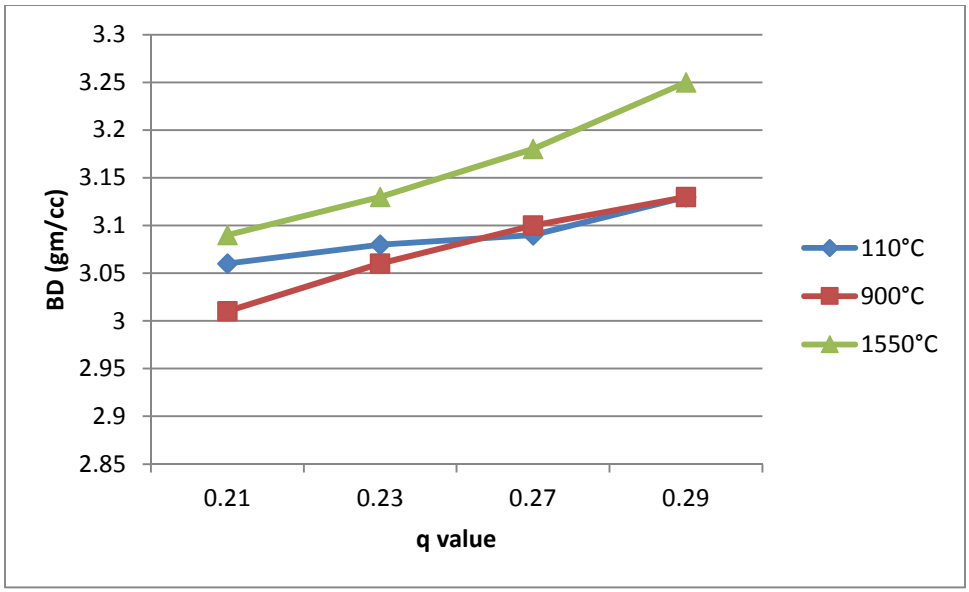


Figure 5.3:-Plot of Bulk Density vs. Q value compositions containing only WTA fine and RA and sol only

Q Value	BD (gm/cc)		
	110°C	900°C	1550°C
0.21	3.03	3.02	3.04
0.23	3.05	3.06	3.08
0.27	3.06	3.08	3.09
0.29	3.10	3.12	3.16

5.4 Density values of compositions containing WTA fine and RA and sol + water

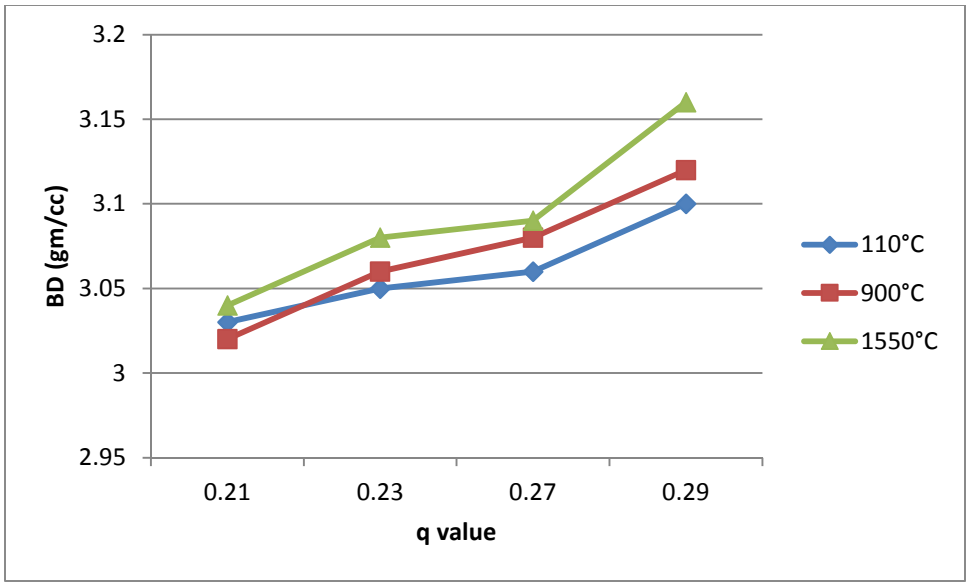


Figure 5.4:-Plot of Bulk Density vs. Q value of compositions containing WTA fine and RA and sol + water

5.2 Permanent Linear change (PLC) on reheating:

In general permanent linear change was found to be negative (shrinkage) at 1550oC for all the types of compositions, containing WTA or WTA with RA, only sol or sol with water, due to sintering of the compositions at high temperatures. But at 900oC there was some variation from batch to batch. But for all the different types of compositions, increase in q values was found to resulted in a reduced extent of shrinkage due to lesser content of fines in the compositions.

Q Value	PLC (%)	
	900°C	1550°C
0.21	0.01	-0.82
0.23	0.02	-0.62
0.27	0.02	-0.55
0.29	0.05	-0.41

5.5 PLC values of compositions containing only WTA fine and sol only

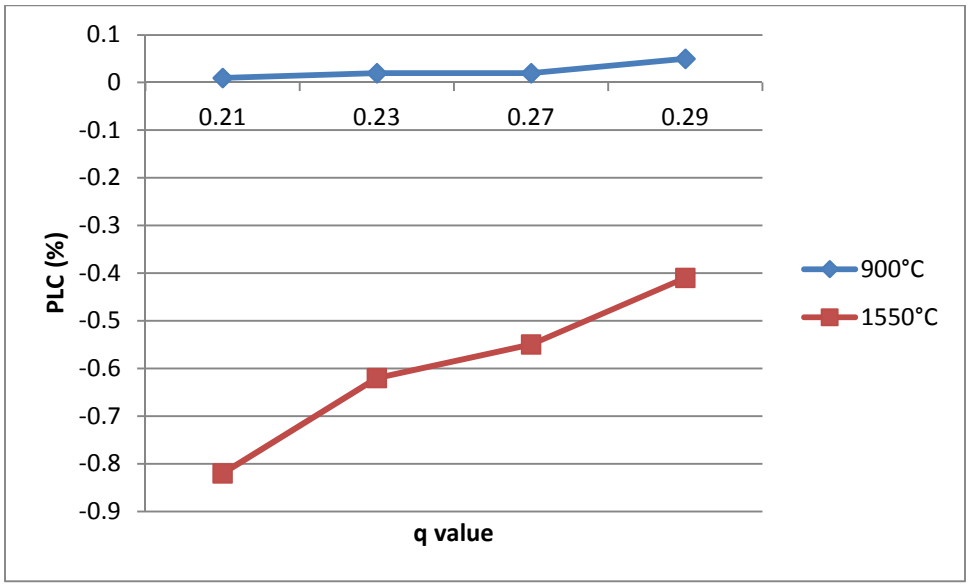


Figure 5.5 Plot of Permanent Linear Change vs. Q value of compositions containing only WTA fine and sol only

In figure 5.5 we see that value of PLC is +ve when sample is fired at 900°C it is due to pores removal process of sample. But PLC is –ve in case of firing at 1550°C , it is due to mullite

formation, due to which some amount of expension is there in the sample. As q value increases PLC value seems to be increasing.

Q Value	PLC (%)	
	900°C	1550°C
0.21	-0.09	-0.68
0.23	-0.04	-0.65
0.27	-0.01	-0.61
0.29	0.03	-0.46

5.6 PLC values of compositions containing only WTA fine and sol+water

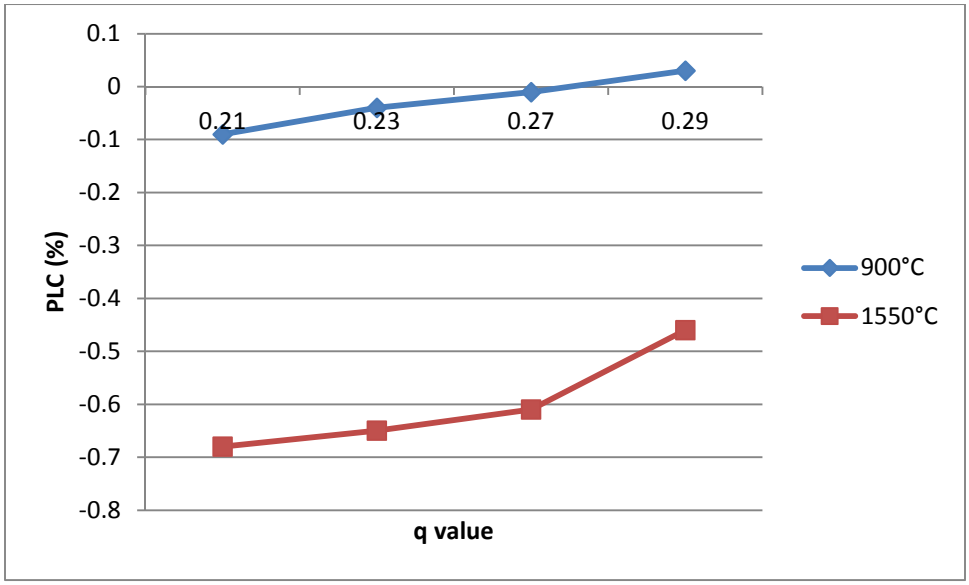


Figure 5.6 Plot of Permanent Linear Change vs. Q value of compositions containing only WTA fine and sol+water

Q Value	PLC (%)	
	900°C	1550°C
0.21	-0.02	-0.87
0.23	-0.02	-0.66
0.27	0.02	-0.53
0.29	0.04	-0.49

5.7 PLC values of compositions containing only WTA fine + RA and sol only

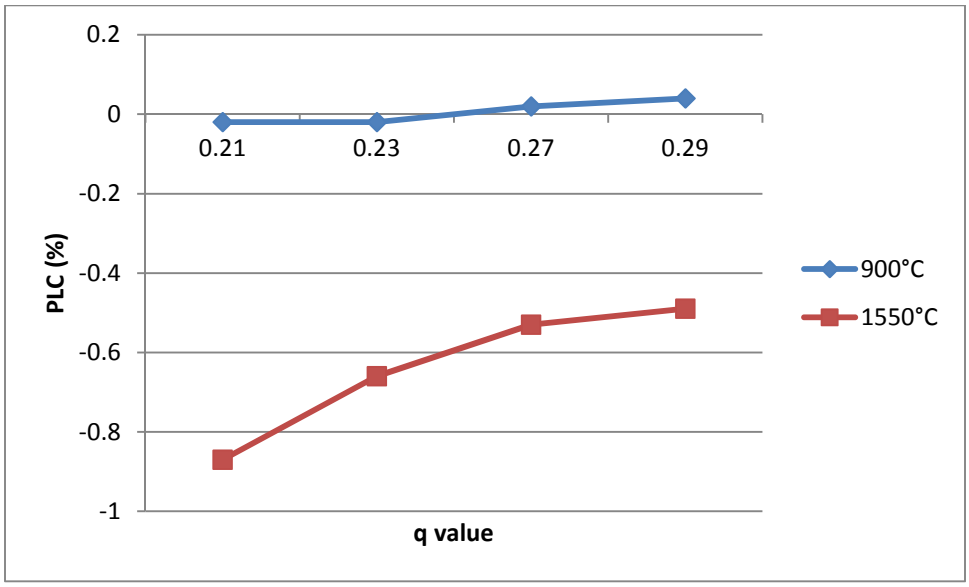


Figure 5.7 Plot of Permanent Linear Change vs. Q value of compositions containing only WTA fine + RA and sol only

Q Value	PLC (gm/cc)	
	900°C	1550°C
0.21	-0.02	-0.76
0.23	-0.01	-0.73
0.27	-0.01	-0.64
0.29	0.03	-0.59

5.8 PLC values of compositions containing only WTA fine +RA and sol+water

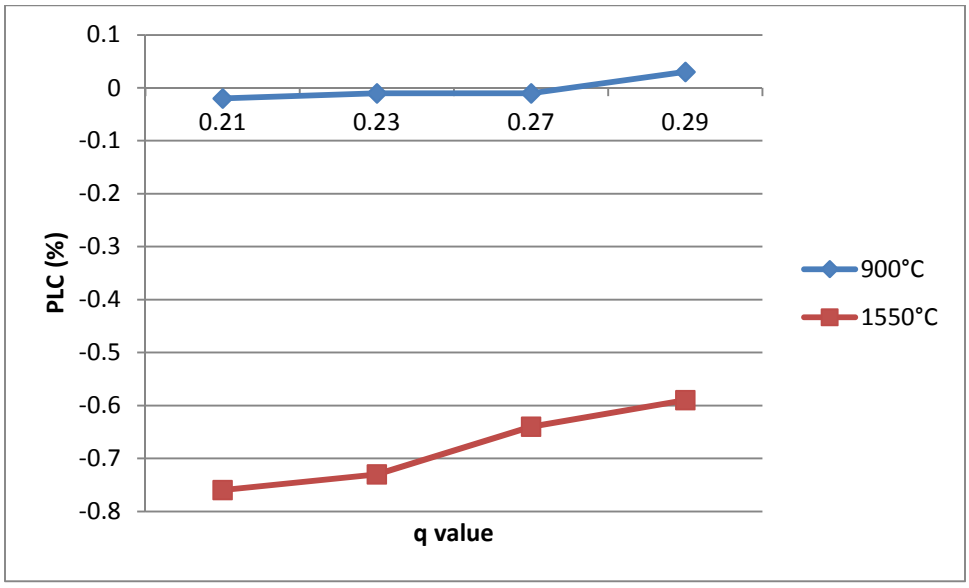


Figure 5.8 Plot of Permanent Linear Change vs. Q value of compositions containing only WTA fine +RA and sol+water

5.3 Cold Crushing Strength

Strength values was found to be dependent on q values of the composition, temperature of heat treatment, content of so, or sol with water and WAT or WTA + RA as fines. In general increasing temperature resulted in an increase in strength but the rise in strength was very prominent when the compositions were fired at 1550oC. Increasing q values were found to increase the strength values, this may be associated with the increase density values and corresponding packing. Also addition of RA replacing partially the WTA fines was found to improve the strength values mainly due to better flowability, compaction and densification. But use of sol with water and bonding material instead of pure sol was found to deteriorate the strength values due to increase extent of porosity resulted in from free water and reduced amount of active fine silica particles coming from sol.

Q Value	CCS (Kg/cm^2)		
	110°C	900°C	1550°C
0.21	324	478	1211
0.23	354	488	1430
0.27	358	542	1544
0.29	416	578	1962

5.9 CCS values of compositions containing only WTA fine and sol

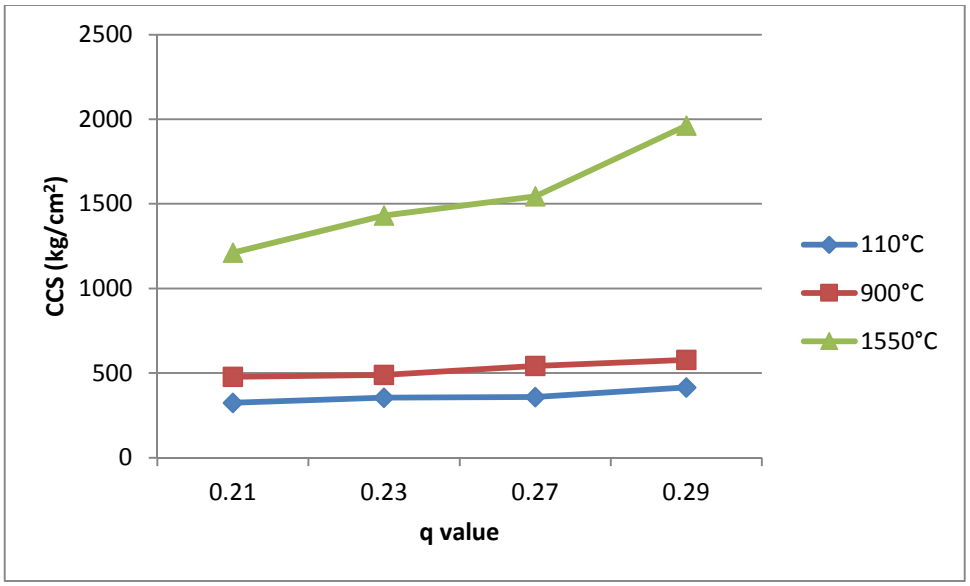


Figure 5.9:- Plot of Cold Crushing Strength vs. Q value of compositions containing only WTA fine and sol

Q Value	CCS (Kg/cm^2)		
	110°C	900°C	1550°C
0.21	261	298	1039
0.23	259	362	1288
0.27	289	414	1467
0.29	346	470	1662

5.10 CCS values of compositions containing only WTA fine and sol+ water

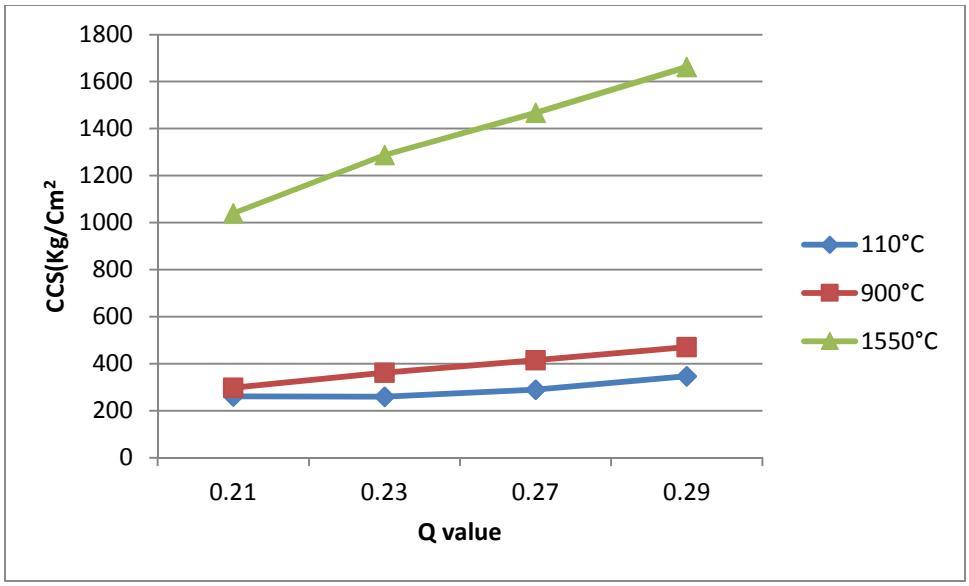


Figure 5.10:- Plot of Cold Crushing Strength vs. Q value of compositions containing only WTA fine and sol+ water

Q Value	CCS(kg/cm²)		
	110°C	900°C	1550°C
0.21	429	787	1032
0.23	443	806	1257
0.27	465	701	1658
0.29	493	652	1913

5.11 CCS values of compositions containing WTA fine + RA and only sol

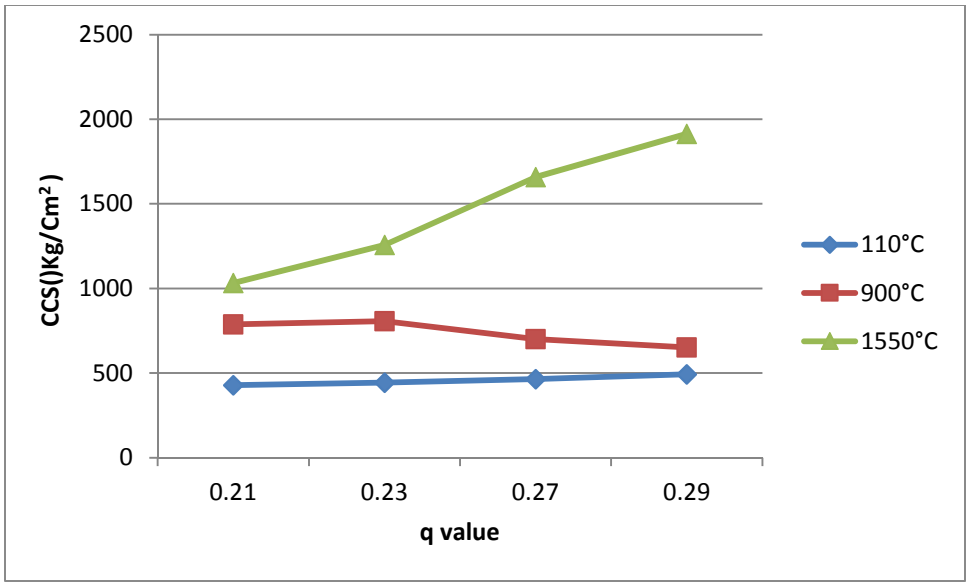


Figure 5.11:- Plot of Cold Crushing Strength vs. Q value of compositions containing WTA fine + RA and only sol

Q Value	CCS(kg/cm^2)		
	110°C	900°C	1550°C
0.21	328	306	949
0.23	345	356	1134
0.27	368	391	1305
0.29	393	510	1616

5.12 CCS values of compositions containing WTA fine + RA and sol+water

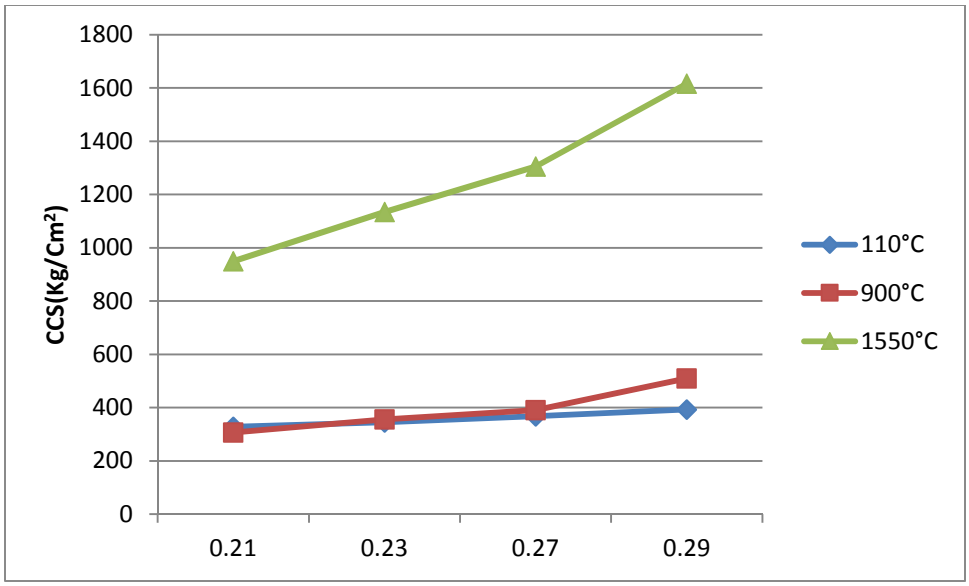


Figure 5.12:- Plot of Cold Crushing Strength vs. Q value of compositions containing WTA fine + RA and sol+water

5.4 CMOR VALUES

Cold MOR values were found to vary similar to that of the CCS variations. CMOR values were found to be dependent on q values of the composition, temperature of heat treatment, content of so, or sol with water and WAT or WTA + RA as fines. In general increasing temperature resulted in an increase in strength but the rise in strength was very prominent when the compositions were fired at 1550oC. Increasing q values were found to increase the strength values, this may be associated with the increase density values and corresponding packing. Also addition of RA replacing partially the WTA fines was found to improve the strength values mainly due to better flowability, compaction and densification. But use of sol with water and bonding material instead of pure sol was found to deteriorate the strength values due to increase extent of porosity resulted in from free water and reduced amount of active fine silica particles coming from sol.

Q Value	MOR (Kg/cm^2)		
	110°C	900°C	1550°C
0.21	40	90	311
0.23	59	75	336
0.27	65	111	349
0.29	72	133	367

5.13 CMOR values of compositions containing WTA fine and sol only

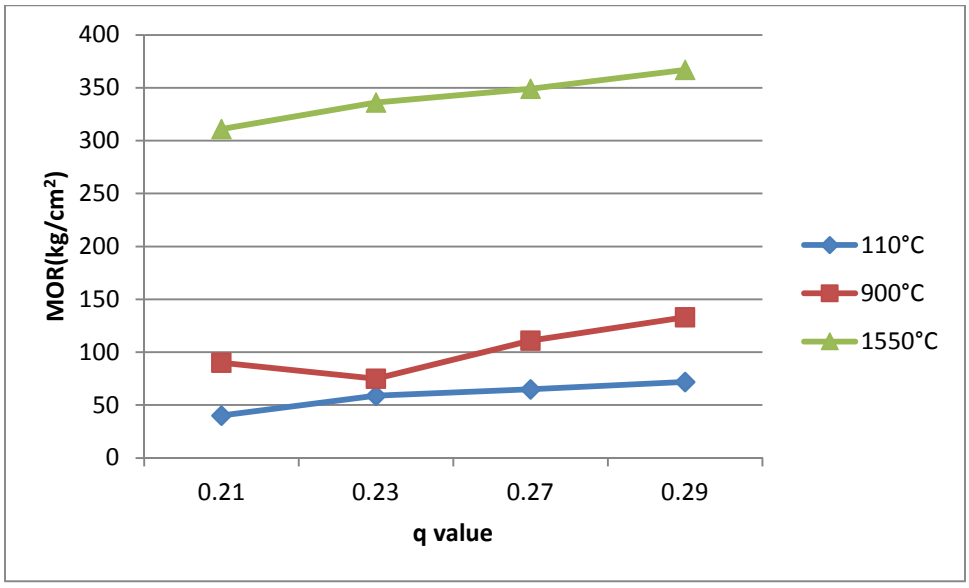


Figure 5.13:- Plot of Modulus of rupture vs. Q value of compositions containing WTA fine and sol only

Q Value	MOR (Kg/cm^2)		
	110°C	900°C	1550°C
0.21	31	75	285
0.23	51	89	294
0.27	55	99	315
0.29	61	109	327

5.14 CMORvalues of compositions containing WTA fine and sol + water

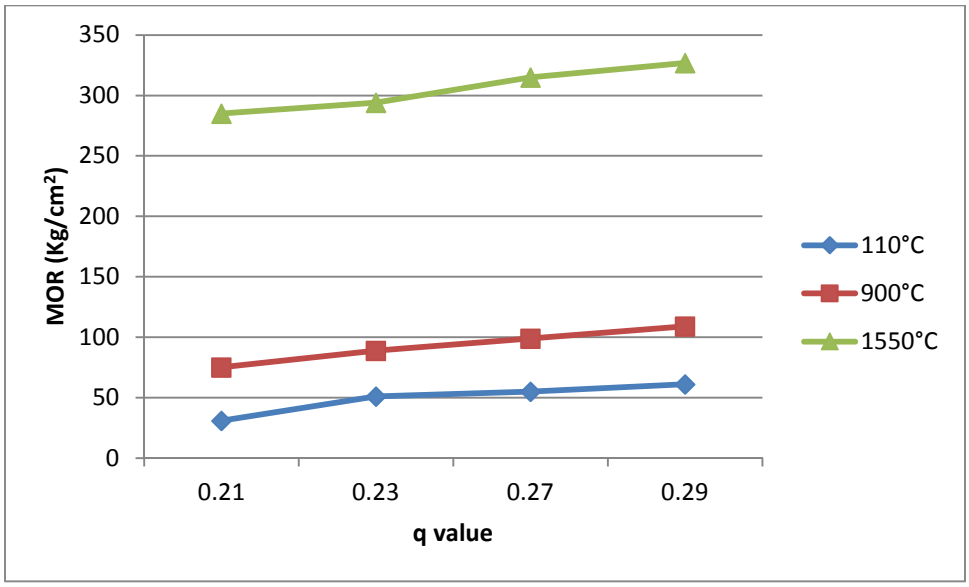


Figure 5.14:- Plot of Modulus of rupture vs. Q value of compositions containing WTA fine and sol + water

Q Value	MOR(kg/cm^2)		
	110°C	900°C	1550°C
0.21	62	161	341
0.23	62	174	370
0.27	74	187	403
0.29	78	204	393

5.15 CMOR values of compositions containing WTA fine + RA and sol only

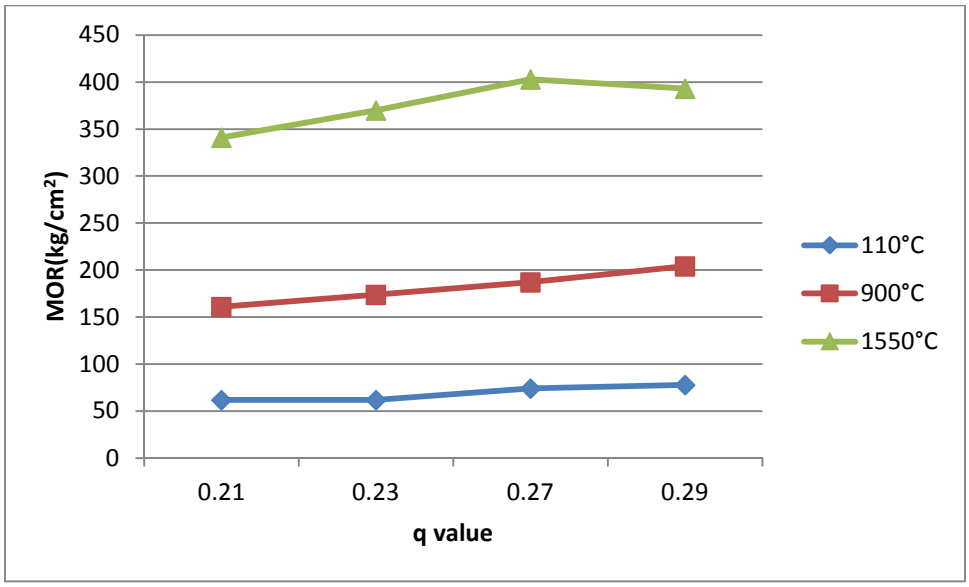


Figure 5.15:- Plot of Modulus of rupture vs. Q value of compositions containing WTA fine + RA and sol only

Q Value	MOR (Kg/cm^2)		
	110°C	900°C	1550°C
0.21	37	90	313
0.23	41	96	346
0.27	47	103	364
0.29	50	123	377

5.16 CMOR values of compositions containing WTA fine + RA and sol + water

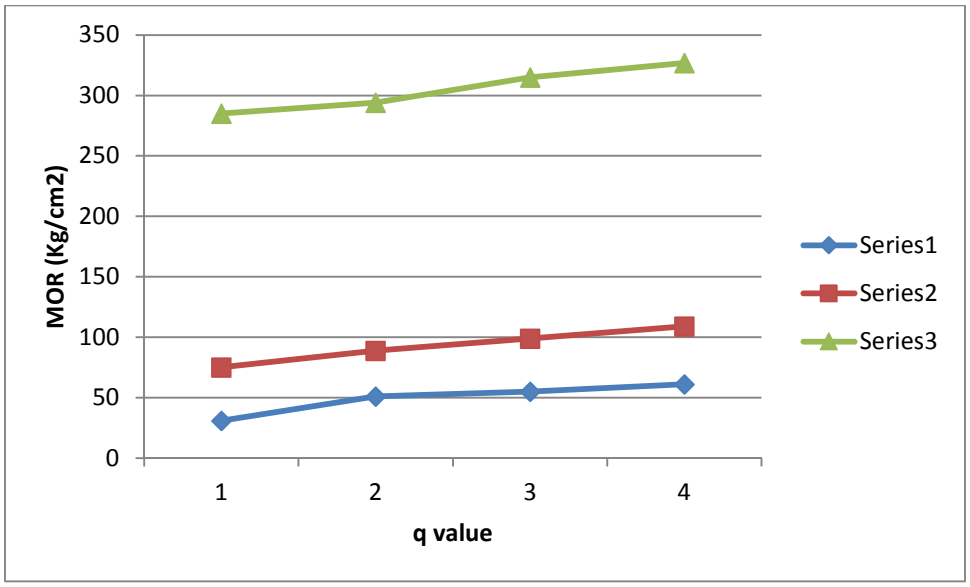
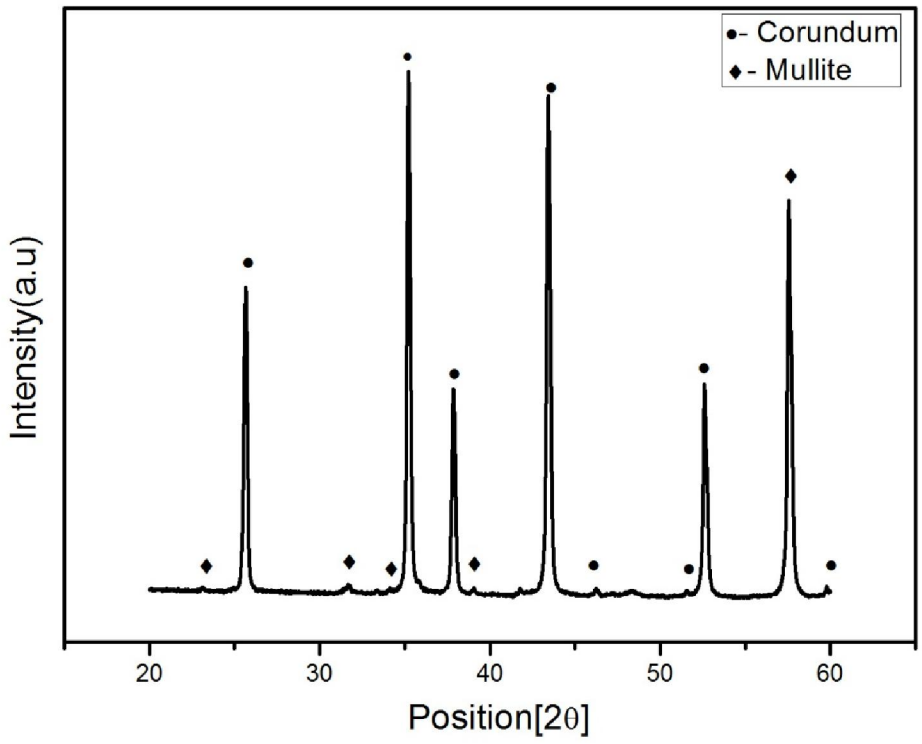


Figure 5.16:- Plot of Modulus of rupture vs. Q value of compositions containing WTA fine + RA and sol + water

5.5 PHASE ANALYSIS by XRD:

The X-ray diffraction method was used for determining the phases in the castable fired at 1550 °C. The castable was crushed and grounded to very fine powders using a mortar pestle and then subjected to XRD for identification of the phases after sintering. The X- Ray data was plotted using the Expert Pro software and the peaks were matched with JCPDS software to get the phases present.



By analyzing above figure we can easily know that there is mullite phase present in the sample which is of q=0.21, 0.23, 0.27, 0.29. All samples were analyzed gave same plot because and each contained mullite phase. This phase is the result of reaction of alumina and silica sol at higher temperature.

CHAPTER 6

SUMMERY & CONCLUSION

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- Resent result shows that q value should be between 0.2 to 0.3 because in this range good flow value and good properties can be obtained for castable [6].
- Self-flowing nature was obtained for q values 0.21 and 0.23 and vibratable castables were developed for q values 0.27 and 0.29 for both WTA fines and reactive alumina containing compositions.
- Reactive alumina, being a fine alumina with controlled particle size distribution, required less amount of water and resulted better flow values.
- Very high density and strength values for fired samples were obtained in the compositions.
- Reaction between mullite phase formation is the reason for high strength development at higher temperatures.
- The study is based on the incorporation of theoretical concept in already practiced commercial product of high alumina low cement castable refractory.
- Incorporation of theoretical concepts in commercial products will improve its performance in Iron and Steel industries and other high temperature applications.
- Both self-flowing and vibratable castables are designed and developed using controlled particle size distribution through theoretical distribution coefficients. The study shows improved characteristics of the developed products for practical applications.
- All the raw materials and additives used for the study are being practiced commercially and enhance the applicability of the study for practical use.
- After studying all the result of q values it can be suggested that by using q value 0.27 we can get better result as far as vibro castable is concerned.

CHAPTER 7

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